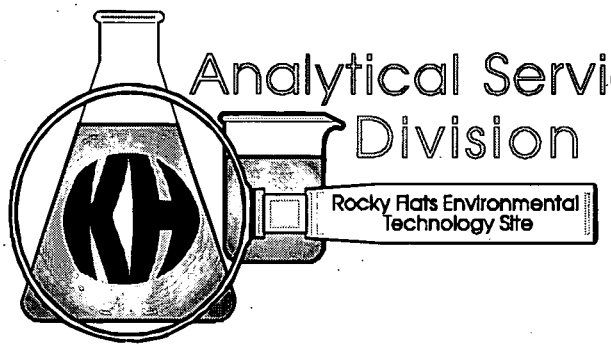


VERIFICATION AND VALIDATION GUIDELINES

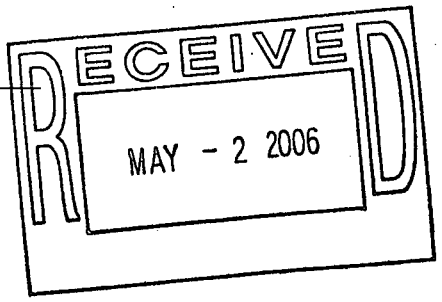
FOR

PCB/PESTICIDES

DA-SS03-v3

Effective Date: October 1, 2002

Approved: E A Bowsky
Analytical Services Division



ADMIN RECORD

Reviewed For Classification
By: Roger S. Cichorz U/NU
ASD Project Lead – T130C
Date: June 4, 2002

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V & V GUIDELINE CHANGE DESCRIPTION FORM

Instructions: Replace Version 2 with Version 3

Guideline: DA-SS03	**Version:** 3	**Originator:** Ed Brovsky
Description: Verification and Validation Guidelines for PCB/Pesticides.		

Section No.	Change Description
N/A	New version and Effective date
Introduction	A new introduction was written to incorporate the BOA SOW rather than PSA Modules.
Entire Document	For clarity, change bars appearing on a Section Title indicate changes to the entire Section.
Entire Document	References to the BOA SOW and the RFETS BOA Implementation document GR03, are incorporated throughout the document. References to PSA Modules were eliminated. References to Module Specific Verification and Validation (V & V) Guidelines were replaced with Analytical Specific V & V guidelines.
Data Review Checklist	All references to the Data Review Checklist and its examination were removed from the Guidelines.
Entire Document	All actions that involve Reason Codes 801, or 803 were revised to include an NCN be issued to request missing, incomplete data, or corrected data. The action requires the discontinuation of further assessment until corrected data is received and the action also requires a comment in the DQA Report identifying the request for missing or corrected data.
2.13	A section was added for TCLP assessment.

1. PURPOSE AND INTRODUCTION

This document presents those data assessment steps which are unique to PCB/Pesticides Analyses. This Analytical Specific document is to be used in conjunction with DA-GR01, "General Guidelines for data Verification and Validation.

The purpose of this document is to provide guidance in the completion of Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division Data Assessment Process as described in DA-GR01.

This version of DA-SS02 is applicable to PCB/Pesticide Sample Data Packages generated under the National Basic Ordering Agreement (BOA) Statement of Work (SOW) and the Rocky Flats Environmental Technology Site (Site) BOA Implementation Requirements documents, GR03 & GR04.

2. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to PCB/Pesticide analyses. They are to be used in conjunction with the general instructions for Verification and Validation found in Analytical Services Division's General Guidelines for Verification and Validation, DA-GR01.

2.1. Chain of Custody, Holding Times, and Sample Preservation

Review Items: COC, Laboratory Sample Receiving Documentation, Cover Page Comments, Sample Case Narrative, raw data, data summary forms, and sample preparation/extraction log.

Objective: The objective is to ascertain the validity of results based on the method required holding times, sample preservation, and the continuity of sample custody.

Source: BOA Attachment 1, § 3.1.2, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Determine if the samples were properly preserved prior to laboratory sample receipt using the criteria provided in Table 1a and Table 1b.

Action 1: If samples were not maintained at 4°±2° C prior to receipt by the laboratory, do not qualify the sample results. However, comment and assign the reason code **[703]** to all applicable samples.

Item 2: Determine if samples were properly preserved after sample receipt.

Action 2: If documentation specifically indicates sample preservation was not maintained after sample receipt, but prior to analysis, issue a Non-Compliance Notification (NCN) requesting a corrective action to prevent recurrence and qualify all results as estimated **[J 201]**.

Item 2: Determine the actual analysis and preparation holding times by comparing the preparation and analysis dates on the raw data and the sample collection date on the COC. If the actual holding time is greater than the maximum allowable holding time identified in Table 1a or Table 1b, use the following actions to qualify all applicable data:

Action 3a: Qualify all positive results as estimated (J) if the actual holding time was greater than the maximum holding time. Assign code **[J 101]** if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code **[J 701]**.

Action 3b: Qualify all non-detected results as estimated (UJ) if the actual holding time was greater than the maximum holding time but less than two times the maximum holding time. Assign code **[UJ 101]** if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code **[UJ 701]**.

Action 3c: Qualify all non-detects as rejected (R) and all detects (J) if the actual holding time was greater than two times the maximum holding time. Assign reason code **[R/J 102]** if the hold time violation is attributed to the lab. If the hold-time violation is not attributed to the laboratory, assign reason code **[R/J 702]**.

Note: Code 701 will apply when samples are received after holding times are expired, or if samples are received after 50% of the holding time has passed.

Table 1a HOLDING TIME AND PRESERVATION CRITERIA

Matrix	Extraction Holding Time (maximum)	Analysis Holding Time (maximum)	Preservation
Water	7 days	40 days	Storage at 4°C
Soil	14 days	40 days	Storage at 4°C

NOTE: The holding time is based on the date when collection was completed, rather than verified time of sample receipt (VTSR).

Table 1b TCLP EXTRACT HOLDING TIME AND PRESERVATION FOR PESTICIDES/HERBICIDES

Holding Time (Days)			Preservation	
TCLP Extraction	Extract Preparation	Extract Analytical	Non-Aqueous Matrix	Aqueous Matrix
14	14	40	Storage at 4°C	Storage at 4°C

2.2. Sample Data Package Narrative

Review Items: Sample case narrative.

Objective: Review the narrative for compliance to requirements and for information useful for validation of data.

Source: GR03 § 3.2, BOA Attachment 1, § 3.1.6.2

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that the SDP Narrative is present and includes the following as applicable:

- Procedures and/or Standard Method reference for preparation and analysis.
- Descriptions of significant technical difficulties encountered in preparing and analyzing the samples.
- Justification of all dilutions.
- Explanations of any QC deficiencies, missed holding times, or inability to achieve the required detection limits (RDLs).
- Reasons for reanalysis, reanalysis Analytical Batch Identifications Numbers, and a synopsis of the reanalysis Analytical Batch QC Assessment.
- Explanations and descriptions of all deviations from routine protocols, including deviations from approved standard operating procedures (SOPs), detection limit modifications, etc. If it was necessary to contact the CTR for instructions due to the nature of the deviation, the laboratory shall document those instructions in the narrative.

Action 1: If any of the above items are non-compliant, do not qualify the results, comment and include the reason codes **[227]** and/or **[805]** as appropriate. Use professional judgement to determine if the issuance of a NCN is warranted.

2.3. Surrogate Recovery

Review Items: Forms 2E/2F or equivalent, Form 6E or equivalent, Form 8D or equivalent, sample preparation/extraction log, sample chromatograms and integration reports.

Objective: To assess laboratory performance based on the results of surrogate spike recoveries. Evaluate the results of the surrogate spikes. Laboratory performance on individual samples is established by means of spiking samples with surrogate compounds prior to extraction and analysis to determine surrogate spike recoveries. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results are frequently subjective and demand analytical experience and professional judgment.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 2E/2F are present.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that surrogate recoveries are reported for all sample, spike, and blank analyses.

Action 2: If required surrogate recoveries are not provided, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Check that the surrogate percent recoveries (%R) are within the limits of Table 2.

SW-846

For SW-846, the laboratory may use the recovery from either surrogate to fulfill the %R requirement. Therefore, no qualification of the data is necessary if one of the two surrogates is inside the control limits.

CLP Method

For CLP, both surrogates are necessary to fulfill the %R requirement. In general, no action is taken unless two of the four recoveries (%Rs) per sample are outside the control limits. However, comment and assign reason code **[142]** to all applicable data.

If two or more of the surrogates exceed the control limits for %R(s) as indicated above (SW-846 and CLP), qualify as follows:

Action 3a: If the sample %R(s) is greater than the control limits, estimate **[J 142]** positive results.

Action 3b: If the sample %R(s) is less than the control limits but greater than or equal to 10%, estimate **[J 142]** positive results and **[UJ 142]** non-detected results.

Action 3c: If the sample %R(s) is greater than zero but less than 10%, estimate **[J 142]** positive results and reject **[R 142]** non-detected results.

Action 3d: For CLP only, if one %R is greater than the control limits and another %R is less than the control limits but greater than or equal to 10%, estimate **[J 142]** positive results and **[UJ 142]** non-detected results.

Table 2 SURROGATE CONTROL LIMITS

Method	Surrogate Compounds	Control Limits
CLP-SOW	Tetrachloro-m-xylene, Decachlorobiphenyl	30-150% (water & soil)
SW-846 8081A/8082	Tetrachloro-m-xylene, Decachlorobiphenyl	Laboratory-determined

Item 4: Check that surrogate retention times (Form 8D) are within the retention time limits provided by the laboratory.

Action 4: If surrogate retention times are outside of the retention time limits, use professional judgment to qualify the data. Consider how much the retention time varied, presence/absence of positive results, MS/MSD recoveries (demonstrates ability to identify positive results), etc.

Dilutions

Compounds reported from the diluted sample will be assessed using the surrogate recoveries from the diluted sample. No action should be taken if a surrogate recovery cannot be reported because of sample dilution. However, professional judgment may be used to warrant qualification.

Item 5: If no surrogate recovery is reported due to dilution, determine if the dilution factor was high enough to justify the surrogates being diluted out.

Action 5: Comment that surrogates were diluted out of the sample and no action was taken. Assign code **[142]** to all sample results associated with diluted surrogates.

Evaluation: ***The following items apply to validation only:***

Item 6: Check chromatograms and quantitation reports to evaluate the recoveries. Verify at least one surrogate recovery per sample.

Action 6: If calculated recoveries are not within 5% of reported result, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

Item 7: Check raw data for interferences or misidentification when %R values are outside of control limits.

Action 7a: If raw data confirms % R, no action is required.

Action 7b: If raw data indicates misidentification, assign reason code **[804].** Use professional judgment to assign a qualifier based on the severity of the problem.

Item 8: When no sample %R is reported (e.g., D or DIL is reported instead of a percent recovery), examine the sample data to determine if the surrogate may be present but slightly outside its retention time window.

Action 8: If the surrogate can be clearly identified, the surrogate recovery should be recalculated and the recalculated value may be used to qualify the data.

2.4. MS/MSD Recovery

Review Items: Forms 3E/3F or equivalent, Form 6E or equivalent, MS/MSD chromatograms and integration reports.

Objective: To determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 3E/3F are present and that MS/MSD analyses were performed at the required frequency.

Action 1: If forms are not present or were not analyzed at the required frequency, comment that the SDP did not include an MS/MSD. No reason code is applied.

Item 2: Check that the MS/MSD percent recoveries (%R) and relative percent differences (RPD), for only the compounds listed in Table 3, are within the identified limits.

Note: No action is taken on MS/MSD data alone to qualify an entire batch. However, using informed professional judgment the data Reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

Action 2: If MS/MSD recoveries or RPDs are not within the limits of Table 3, comment that limits were not met. Do not qualify, but assign reason code **[231]** to the outlying compound in all associated samples. The data reviewer may use the MS/MSD results in conjunction with other QC criteria to determine if data qualification is warranted.

Table 3 MS/MSD FREQUENCY AND CONTROL LIMITS

Spiking Compound	CLP-SOW %R Limits[RPD Limit]		SW-846 8081A %R Limit	SW-846 8082 %R Limit
	Water	Soil	Not specified. Use lab limits.	Not specified. Use lab limits.
gamma-BHC (Lindane)	56-123[15]	46-127[50]		**Note**
Heptachlor	40-131[20]	35-130[31]		• Aroclors 1016/1260 may be used to represent all Aroclors
Aldrin	40-120[22]	34-132[43]		• Must inject other aroclors if found in samples
Dieldrin	52-126[18]	31-134[38]		
Endrin	56-121[21]	42-139[45]		
4,4'-DDT	38-127[27]	23-134[50]		
	Frequency: 1/20 samples		**Frequency: 1/20 samples**	**Frequency: 1/20 samples**

Evaluation: ***The following item applies to validation only:***

Item 3: Calculate at least one %R and one RPD value in the MS/MSD data using the following calculations:

$$\%R = \frac{Found\_Value}{True\_Value} \times 100$$

$$RPD = \frac{|D_1 - D_2|}{\left(\frac{D_1 + D_2}{2}\right)} \times 100$$

where:

D_1 = MS Concentration.

D_2 = MSD Concentration.

Action 3: If the %R or % RPD values cannot be verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

2.5. Sample Results

Review Items: Form 1D or equivalent, Forms 6E/6F or equivalent, Forms 7D/7E or equivalent, Form 8D or equivalent, Forms 10A/10B or equivalent, COC record, extraction logs, sample chromatograms and integration reports, and GC/MS confirmation data (if applicable).

Objective: To determine if false positives (reporting a compound present when it is not) or false negatives (not reporting a compound that is present) were reported by evaluating qualitative criteria for compound identification.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Form 1D is present for each sample including method QC.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that significant figures and flagging protocol are as specified in the latest version of CLP.

Action 2: If significant problems exist, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Determine if Forms 1D contain "B" qualifiers.

Action 3a: If "B" qualifiers are present, determine if blank contamination is addressed in the SDP Narrative. If contamination is not addressed, do not qualify the results. Comment and include the reason code **[805].**

Action 3b: If "B" qualifiers are present, proceed with the qualification specified under Blanks.

Retention Time Windows

Item 4: Confirm positive results by reviewing Forms 10A/10B to ensure that all positive results were within the retention time windows (use initial calibration windows for CLP; daily calibration windows for SW-846).

Action 4a: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are met but the compound is reported as non-detected, the result may be a false negative. Use professional judgment either to quantitate and report the positive result or to reject **[R 145]** the non-detected result.

Action 4b: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, use professional judgment to qualify non-detected results **[U 145]** or reject **[R 145]** the positive result.

Confirmation

Item 5: Determine if the percent difference in a positive concentration between the two columns is met.

Action 5: If the difference in a positive concentration between both columns is greater than 25% D, then qualify the affected compound as estimated **[J 131]**.

Evaluation: ***The following items apply to validation only:***

Confirmation

Item 6: Verify the transcription of all results from the chromatogram and integration report to the Form 1D and Forms 10A/10B.

Action 6: If reviewed results are not transcribed accurately, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 7: Verify that primary and secondary chromatograms are present for all samples analyzed by CLP and for those samples with positive results analyzed by SW-846.

Action 7: If confirmation data are not provided and a positive result (which may or may not have been reported) is evident in the primary data, reject **[R 145]** the result (which is either a reported positive result or a non-detected result).

Retention Time Windows

Item 8: Further review positive results by reviewing Forms 10A/10B against the sample chromatograms and integration reports. For multi-component compounds, the retention times and relative peak height ratios or major peaks should be compared to the appropriate standard chromatograms.

Action 8a: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are met but the compound is reported as non-detected, the result may be a false negative. Use professional judgment either to quantitate and report the positive result or to reject **[R 145]** the non-detected result.

Action 8b: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, use professional judgment to change the result to non-detected **[U 145]** at the MDL or reject **[R 145]** the positive result.

Item 9: If retention time windows are not provided, evaluate the samples based upon the retention time shifts of the target compounds in the calibration standards, the retention time shifts of the surrogates in the calibration standards and samples, the abundance of peaks in the samples above the MDL, the number of target compounds under consideration, etc.

Action 9: Use professional judgment to qualify the data as valid or rejected **[R 199]**.

Item 10: If multi-component target compounds exhibit marginal pattern-matching quality, professional judgment should be used to determine if this is due to environmental "weathering" (i.e., degradation of the earlier eluting peaks relative to the later eluting peaks).

Action 10: If the presence of a multi-component compound is strongly suggested, results should be reported as presumptively present **[NJ 199]**.

Cleanup

Item 11: Verify that cleanup techniques were employed for samples with interferences present on the chromatography.

Action 11: If no cleanup techniques were employed do not qualify any data. Comment and assign reason code **[199]** to all applicable data.

Interference

Item 12: The Reviewer should be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carry-over is a possibility and should use judgment to determine if carry-over has occurred.

Action 12a If interference/carry-over is causing identification problems of reported positive or non-detected target compounds, professional judgment should be used to evaluate the severity of the interference and to apply one of the following actions: estimate **[J 199]** the positive result, estimate **[UJ 199]** the

non-detected result, reject **[R 199]** the positive result, or reject **[R 199]** the non-detected result.

Action 12b If the detection of a high level or multi-component target compound interferes with the detection of another target compound, use professional judgment to raise the MDL to the lower value of the two columns and report that MDL as either valid or estimated **[J 199]**. (This is most applicable when it is evident that the laboratory has performed similar action on other sample results.)

Gas Chromatography/Mass Spectrometry

Item 13: Verify that GC/MS confirmation was performed for pesticide concentrations exceeding 10 ng/uL (CLP only) in the sample extract.

Action 13: If not, comment and assign reason code **[199]** to all applicable data.

2.6. Compound Quantitation and RDLs

Review Items: Form 1D or equivalent, Forms 6E/6F or equivalent, Form 8D or equivalent, COC record, sample preparation/extraction logs, sample chromatograms and integration reports.

Objective: To ensure that the reported quantitation results and detection limits are accurate.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Using the Line Item Code from the COC record, determine if the detection limits reported on Form 1D match the required detection limits (RDLs) listed in Attachment K to BOA Attachment 1, GR03, GR04, or other applicable Statement of Work (SOW). Note that dilutions, percent solids, and extraction steps will impact the final RDLs reported.

If RDLs on Form 1D do not meet those required by the Line Item Code requested, check the RIN file for additional information, which may explain the deviation.

Action 1: If an explanation is not found, use professional judgement to qualify non-detected results with reason code **[213]**.

Item 2: Verify that B qualifiers are added to all positive sample results for compounds that are associated with contaminated blanks.

Action 2: If non-compliant, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Evaluate Forms 1D to ensure that no "E" qualifiers are present. If "E" qualifiers are present, ensure that another Form 1D with a diluted sample analysis is present in the data package.

Action 3: If "E" qualifiers are present and there is not a Form 1D with a diluted sample analysis, comment and estimate **[J 148]** the positive "E" result.

Note: Generally, the analysis with the lower reporting limits are used with the exception of results that exceed the calibration range. Only compounds that originally exceeded the calibration range are reported from the dilution.

Item 4: Ensure that required dilutions are addressed in the SDP Narrative.

Action 4: If not addressed, do not qualify the results. Comment and include the reason code **[805].**

Item 5: Determine from the Form 1B/1C the compounds that were outside the upper half of the calibration range prior to dilution, but fall within the upper half of the calibration range after dilution.

Action 5a: Assign reason code **[155]** only to the data points that meet the above criteria. Do not assign any qualifier to these data points. Any data qualification will be assigned to the data point reported from the dilution.

Action 5b: If the diluted sample analysis fails to keep the response of the major constituents in the upper half of the calibration range, use professional judgment to qualify the data. At a minimum, comment and assign reason code **[252]** to all applicable data.

Evaluation: ***The following items apply to validation only:***

Item 6: Verify that responses for target compounds and standard peaks were measured consistently (i.e., all values were determined by either integrated areas or peak heights, not both).

Acton 6: If the target compound and standard peaks were not measured consistently, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Calculations

Item 7: Compare integration reports, chromatograms, sample preparation/extraction logs, dilutions, and cleanups to the reported sample results.

Action 7: If significant problems exist, or if there are insufficient data to verify calculations, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 8: Examine the raw data to verify the correct calculation of one positive result per sample using the following equations:

Note 1: If first-order linear regression was used for quantitation, sample concentration must be calculated from the equation of

the line via a calculator. Follow the appropriate instructions for linear regression in the calculator literature.

Note 2: If second-order linear regression was used for quantitation, sample concentration must be calculated from the equation of the line provided by the laboratory.

External Standard Technique

$$\frac{ug}{Kg} \text{ or } \frac{ug}{L} = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times \left(\left[V_s\right] \; or \; \left[W \times P\right]\right)}$$

where:

A_x = Response for the analyte in the sample, using peak area or height
A = Amount of standard injected, ng
V_t = Volume of total extract, uL
D = Dilution factor
A_s = Response for external standard, using same units as A_x
V_i = Volume of extract injected, uL
V_s = Volume of water extracted or purged, mL
W = Weight of soil extracted or purged, g
P = Percent Solids/100

Internal Standard Technique

$$\frac{ug}{Kg} \text{ or } \frac{ug}{L} = \frac{A_x \times I_{is} \times D}{A_{is} \times RF \times \left(\left[V_s\right] \; or \; \left[W \times P\right]\right)}$$

where:

A_x = Response for the analyte in the sample, using peak area or height
I_{is} = Amount of internal standard added to volume purged or to extract, ng
D = Dilution factor
A_{is} = Response for the internal standard, using same units as A_x
RF = response factor for analyte, as determined below
V_s = Volume of water extracted or purged, mL
W = Weight of soil extracted or purged, g
P = Percent Solids/100

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Response for the characteristic ion for the analyte to be measured, units area counts
C_{is} = Concentration of the internal standard, ug/L

A_{is} = Response for the characteristic ion for the internal standard, units area counts

C_s = Concentration of the analyte to be measured, ug/L

Action 8: If the concentrations are not verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.7. Calibration

Review Items: Forms 6D-J or equivalent, Forms 7D/7E or equivalent, sample and standard chromatograms and integration reports.

Objective: To determine if the instrument calibration is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration verification documents satisfactory performance of the instrument over specific time periods during sample analysis.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Resolution Check (CLP) [Pesticide Only]

Item 1: Use Form 6G to verify that the resolution criterion between two adjacent peaks for the required compounds in the Resolution Check Mixture is ≥60%.

Action 1a: If the resolution criterion is not met, quantitative and qualitative results may not be accurate. Estimate **[J 170]** detected target compounds that were not adequately resolved.

Action 1b: Use professional judgment to reject **[R 170]** non-detects with retention times in the region of coelution, depending upon the extent of the problem.

Performance Evaluation Mixture (PEM) [Pesticide Only]

Item 2: Use Form 6H to verify that all peaks in all Performance Evaluation Mixture (PEM) analyses are ≥90% resolved.

Action 2a: If PEM resolution criteria are not met, quantitative and qualitative results may not be accurate. Estimate **[J 170]** detected target compounds that were not adequately resolved.

Action 2b: Use professional judgment to reject **[R 170]** non-detects with retention times in the region of coelution, depending upon the extent of the problem.

Item 3: Verify that the absolute retention times of each single component pesticide and surrogate in all PEM analyses are within the specific retention time windows.

Action 3a: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are met but the compound is reported as non-detected, the result may be a false negative. Use professional judgment either to quantitate and report the positive result or to reject **[R 145]** the non-detected result.

Action 3b: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, use professional judgment to qualify non-detected results **[U 145]** or reject **[R 145]** the positive result.

Percent Breakdown (Pesticide Only)

Item 4: Verify that individual breakdowns for 4,4'-DDT and endrin meet the criteria contained in Table 4.

Action 4a: If 4,4'-DDT breakdown exceeds criteria or is not performed qualify as follows:

- Estimate **[J 147]** positive results for 4,4'-DDT.
- If 4,4'-DDT was not detected but 4,4'-DDD and 4,4'-DDE were detected, reject **[R 147]** the non-detected result for 4,4'-DDT.
- Qualify as presumptively present at an estimated quantity **[NJ 147]** positive results for 4,4'-DDD and 4,4'-DDE.

Action 4b: If endrin breakdown exceeds criteria or is not performed qualify as follows:

- Estimate **[J 147]** positive results for endrin.
- If endrin was not detected but endrin aldehyde and endrin ketone were detected, reject **[R 147]** the non-detected result for endrin.
- Qualify as presumptively present at an estimated quantity **[NJ 147]** positive results for endrin aldehyde and endrin ketone.

Action 4c: If the combined 4,4-DDT and endrin breakdown (CLP only) is >30.0%, consider the degree of individual breakdown of 4,4'-DDT and endrin and apply qualifiers as described above.

Table 4 PERCENT BREAKDOWN LIMITS

Method	Compound	%Breakdown Limit
CLP	Endrin, 4,4'-DDT	20.0%
	Combined	30.0%
SW-846 8081A	Endrin, 4,4'-DDT	15.0%

Initial Calibration

For SW-846, calibration factors (CFs) may be used for calculation of sample results if they meet the percent relative standard deviation (%RSD) limits contained in Table 5. Otherwise, the laboratory may use a curve for calculation. For CLP, the CFs must be used for calculation and must meet the limits contained in Table 5 (Form 6E).

Item 5: Determine if an inappropriate number of standards were used and an appropriate concentration level was analyzed.

Action 5: If an inappropriate number of standards were used or inappropriate concentration levels were analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code **[168]** to all applicable data.

Percent Relative Standard Deviation

Item 6: Identify those compounds that exceed the %RSD criteria in the associated initial calibration.

Action 6: Estimate **[J 140]** positive results and **[UJ 140]** non-detected results for those compounds whose %RSDs exceed the criteria in the associated initial calibration.

Note: For Aroclors, if multipeak %RSDs are provided then the average %RSD of all the peaks should be used to determine that the %RSD criteria were met.

Table 5 INITIAL CALIBRATION CRITERIA

Method	Compound	# of Standards	Concentration	%RSD Limit
CLP	All single-component compounds	3	5.0-50.0 ng/mL (depends on compound)	20.0%*
SW-846 8081A	All target compounds	5	**Low** Near but above established MDL **All others** Should define the range of the detector used	20.0%
SW-846 8082	All target compounds **Note** • Aroclors 1016/1260 may be used to represent all Aroclors; • Must inject other aroclors if found in samples	5	**Low** Near but above established MDL **All others** Should define the range of the detector used	20.0%

*CLP indicates that up to 2 compounds per column may be less than 30% with no action.

Calibration Curve

Item 7: For the purposes of these guidelines, if a first-order linear regression is used rather than calibration factors, verify that the correlation coefficient (r) for each compound is ≥0.99.

Action 7: Estimate **[J 140]** positive results and **[UJ 140]** non-detected results for those compounds whose correlation coefficient was <0.99 if first-order linear regression was used for quantitation.

Item 8: For the purposes of these guidelines, if a second-order linear regression or a quadratic curve is used rather than calibration factors, verify that the required information is provided to accurately reproduce positive results.

Action 8: Estimate **[J 140]** positive results if the sample results cannot be reproduced using the second-order or quadratic equation provided for the initial calibration.

Continuing Calibration

For SW-846, if CFs are used for calculation of sample results, they must be less than or equal to the percent difference (%D) limits in the table below. For CLP, the CFs must be used for calculation and must be less than or equal to the limits contained in Table 6 (Forms 7D/7E).

Item 9: Determine if the continuing calibration frequency was met and an appropriate concentration level was analyzed.

Action 9: If the continuing calibration frequency criteria were not met or if inappropriate concentration levels was analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code **[168]** to all applicable data.

Percent Difference

Item 10: Identify those compounds that exceed the %D criteria in the associated (bracketing) continuing calibration.

Action 10: Estimate **[J 141]** positive results and **[UJ 141]** non-detected results for those compounds whose %Ds exceed the criteria in the associated (bracketing) continuing calibrations.

Note: For Aroclors, if multipeak %Ds are provided, then average %Ds should be used to determine that the %D criteria were met.

Table 6 CONTINUING CALIBRATION CRITERIA

Method	Standard	Frequency	Concentration	%D Limit
CLP	Alternate PEM/Individual Mix A and B	Every 12 hours and at the end of the analysis sequence	PEM has only one level; Individual A and B midpoint	25.0%
SW-846 Method 8081A	All target compounds	Each working day, every 10 samples, and at the end of the analysis sequence	Mid-level	15.0%
SW-846 Method 8082	All target compounds **Note** Aroclors 1016/1260 may be used to represent all Aroclors	Each working day, every 10 samples, and at the end of the analysis sequence	**Low** Near but above established MDL **All others** Should define the range of the detector used	15.0%

Retention Time: In addition to the criteria presented in Table 6, the daily retention time windows must be met by each subsequent continuing calibration in a sequence.

Item 11: Review Forms 7D/7E to ensure that the retention times of the associated continuing calibration fall within the established retention time windows.

Action 11: If retention times do not fall within established time windows, use professional judgment to tentatively identify and estimate **[NJ 199]** the positive results for the affected compounds.

Evaluation: ***The following items apply to validation only:***

Percent Breakdown

Item 12: Verify at least one % Breakdown value using the following equations:

$$4,4'-DDT = \frac{Peak\ area\left(4,4'-DDD + 4,4'-DDE\right)}{Peak\ area\left(4,4'-DDD + 4,4'-DDE + 4,4'-DDT\right)} \times 100$$

$$Endrin = \frac{Peak\ area\left(endrin\ aldehyde + endrin\ ketone\right)}{Peak\ area\left(endrin\ aldehyde + endrin\ ketone + endrin\right)} \times 100$$

$$Combined\ \%\ Breakdown = \%\ Breakdown\ 4,4'-DDT + \%\ Breakdown\ Endrin$$

Action 12: If the calculation for % Breakdown cannot be verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

Initial Calibration

Item 13: Check the raw data and verify at least one CF per calibration standard. Recalculate at least one average CF and %RSD:

$$CF = \frac{total\ area\ of\ peak}{nanograms\ injected}$$

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{(n-1)}}$$

where:

X_i = Each individual value used to calculate the mean

$\overline{X}$ = The mean of initial calibration factors

n = The total number of initial calibration factors

Action 13: If the calculation for CF or % RSD cannot be verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

Calibration Curve

If first-order linear regression was used for quantitation, verify one correlation coefficient (r) following the appropriate instructions for linear regression in the calculator literature.

If second-order linear regression or quadratic curves were used for quantitation, verify that results are reproducible using the provided second-order equation.

Continuing Calibration

Item 14: Recalculate at least one average CF and %D:

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R_1 = Calibration factor from first analysis.

R_2 = Calibration factor from subsequent analysis.

Action 14: If the calculations for %D, or CF, cannot be verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

Retention Time

Item 15: If continuing calibration retention times are not within their appropriate retention time windows, carefully examine the raw data for false positive or false negative results.

Action 15: Peaks outside the retention time window but shifted in the appropriate magnitude (relative to that of the standard) may be considered acceptable. At a minimum, comment and assign reason code **[804]** to all applicable data.

2.8. Analytical Sequence (CLP)

Review Items: Form 8D or equivalent

Objective: To ensure calibration provides a sound, comparable analytical approach to initial calibration, continuing calibration, and instrument performance.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: For CLP analyses, examine Form 8D or equivalent and determine if the analytical calibration sequence identified in Table 7 is met.

Table 7 ANALYTICAL CALIBRATION SEQUENCE FOR CLP

	1	Resolution Check
	2	PEM
	3	Aroclor 1016/1260
	4	Aroclor 1221
	5	Aroclor 1232
	6	Aroclor 1242
	7	Aroclor 1248
	8	Aroclor 1254
	9	Toxaphene
	10	Low Point Standard A
	11	Low Point Standard B
	12	Midpoint Standard A
	13	Midpoint Standard B
	14	High Point Standard A
	15	High Point Standard B
	16	Instrument Blank
0 hour	17	PEM
	18	First sample
		Samples
12 hours		Last sample
		1st injection past 12 hours = Instrument Blank
		Individual Mix A
		Individual Mix B
		SAMPLES
12 hours		Last Sample
		1st injection past 12 hours = Instrument Blank
		PEM
		etc.

Action 1: If the sequence was not followed as required, determine the severity of the problem and its effect of the data using professional judgment. At a minimum, comment and assign reason code **[168]** to all applicable data.

2.9. Florisil Cartridge Check (CLP)

Review Items: Form 9A or equivalent, Florisil data.

Objective: To ensure pesticide cleanup procedures remove matrix interferences from sample extracts prior to analysis. Florisil cartridge cleanup significantly reduces matrix interference caused by polar compounds. Pesticide cleanup procedures are checked by spiking the cleanup columns and cartridges and verifying the recoveries.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Ensure that all samples are accounted for on one of the Forms 9A

Action 1: If not all of the samples can be accounted for on one of the Forms 9A, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Examine Form 9A to ensure that the recoveries are within the 80-120% recovery limits.

Action 2: If recoveries are outside the limits, determine the severity of the problem and its effect on the data using professional judgment. At a minimum, comment and assign reason code **[211]** to all applicable data.

Evaluation: ***The following item applies to validation:***

Item 3: Examine Form 9A to ensure that the recoveries are within the 80-120% recovery limits. If florisil recoveries are outside the limits, examine the raw data for the presence of polar interferences. Use the presence or absence of polar interferences in qualifying the data using professional judgment.

Action 3: Low recoveries may result in the qualification of data as estimated **[J 211]**. High recoveries may result in the qualification of detected results **[J 211]**.

Note: These items are used to assess the impact of low recoveries on the Form 9B. However, they are not solely used to qualify data.

Item 4: Recalculate 10% of the percent recoveries on Form 9A. Check transcription of the percent recoveries.

Action 4: If the recoveries are not calculated correctly, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.10. Gel Permeation Chromatography (GPC) [CLP]

Review Items: Form 9B or equivalent, GPC data, GPC run logs.

Objective: To ensure pesticide cleanup procedures remove matrix interferences form sample extracts prior to analysis. GPC removes high molecular weight contaminants.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Ensure that all samples are accounted for on one of the Forms 9B.

Action 1: If not all of the samples can be accounted for on one of the Forms 9A, comment and assign reason code **[804]**.

Item 2: Examine Form 9B to ensure that the recoveries are within the 80-110% recovery limits.

Action 2a: If high recovery is reported, estimate **[J 199]** associated positive results for that compound.

Action 2b: If zero recovery is reported, **[R 199]** associated non-detected results for that compound.

Action 2c: If low recoveries are reported, determine the severity of the problem and its effect of the data using professional judgment. At a minimum, comment and assign reason code **[211]** to all applicable data.

Evaluation: ***The following item applies to validation only:***

Item 3: Recalculate 10% of the percent recoveries on Form 9B. Check transcription of the percent recoveries. In the raw data, check that the Aroclor patterns are similar to those of previous Aroclor standards.

Action 4: If the recoveries are not calculated correctly or if the Aroclor patterns are not similar to other Aroclor patterns, assign the reason code **[804]** to all applicable data points. However, do not qualify the data.

Item 4: If GPC recoveries are outside the limits, examine the UV traces, chromatograms, and integration reports for the presence of high molecular weight compounds. Use their presence or absence for help in qualifying the data.

Action 4: Low recoveries may result in the qualification of data as estimated **[J 211]**. High recoveries may result in the qualification of detected results **[J 211]**.

Note: These items are used to assess the impact of low recoveries on the Form 9B. However, they are not solely used to qualify data.

Item 5: Verify that the absolute retention times of each single component pesticide and surrogate in all PEM analyses are within the specific retention time windows.

Action 5a: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are met but the compound is reported as non-detected, the result may be a false negative. Use

professional judgment either to quantitate and report the positive result or to reject **[R 145]** the non-detected result.

Action 5b: If the criteria for positive identification (i.e. peak within its window on both columns or any evident shifts explained) are not met, use professional judgment to change the result to non-detected **[U 145]** at the MDL or reject **[R 145]** the positive result.

2.11. Blanks

Review Items: Form 4C or equivalent, Instrument Blank, Method Blank, and Sulfur Cleanup Blank Forms 1D or equivalent, chromatograms and integration reports.

Objective: To determine the existence and magnitude of blank contamination problems. The criteria for evaluation of laboratory blanks apply to method, instrument, and sulfur cleanup blanks associated with the samples. If problems with any blank exist, all data associated with the blank must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify that Method Blank Summary Forms (4C) are present.

Action 1: If not provided, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Determine if the blank criteria contained in Table 8 are compliant for the given method.

Note: If more than one blank is associated with a sample, qualification should be based upon comparison of the blank with the highest level of contamination.

Table 8 BLANK CRITERIA

Method	Types	Frequency	Criteria
CLP	Method	1/20 samples of similar matrix in each sample delivery group or whenever a sample extraction procedure is performed.	No contaminants should be present in the blanks. Method blanks should be analyzed on each GC system used to analyze that set of associated samples.
	Instrument	Once at least every 12 hours and immediately prior to the analysis of each continuing calibration (either the PEM or Ind. A/B). Following sample analysis which contain an analyte at a high concentration.	The concentration of each target compound in the instrument blank must be less than 0.5 times the RDL for that compound. (For comparing the results , assume that the material in the instrument blank resulted from the extraction of 1 L of water.)
	Sulfur Cleanup	Modified form of a method blank which has undergone sulfur cleanup. One per SDG (if all underwent sulfur cleanup, the method blank satisfies the sulfur blank requirement) or subset of an SDG which has undergone sulfur cleanup.	The concentration of each target compound in the instrument blank must be less the RDL for that compound. The method blanks should be analyzed on each GC system used to analyze that set of associated samples.
SW-846 8081A	Method	A method blank should be extracted with each extraction batch, when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound.	No contaminants should be present in the blanks. The blank samples should be carried through all stages of the sample preparation and measurement steps (i.e., the method blank should be analyzed on the same instrument as the samples).
SW-846 8082	Method	A method blank should be extracted with each extraction batch, when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound.	No contaminants should be present in the blanks. The blank samples should be carried through all stages of the sample preparation and measurement steps (i.e., the method blank should be analyzed on the same instrument as the samples).

Action 2a: If the proper blanks were not analyzed at the appropriate frequency, determine the severity of the problem and its effect on the data using professional judgment. At a minimum, comment and assign reason code **[168]** to all applicable data.

Action 2b: If a target compound is found at any concentration in the blanks but not in the samples, no action is taken.

Action 2c: If a target compound is found in the blanks at any concentration and is also found in the sample, apply the following:

- If the sample concentration is less than 5 times the blank concentration and less than or equal to the RDL, qualify the result as estimated **[JB 249]**.
- If the sample concentration is less than or equal to 5 times the blank concentration and greater than the RDL, qualify the data **[U 249]**.

- If the sample concentration is greater than 5 times the blank concentration and greater than the RDL do not qualify the reported value.

  Note: The Reviewer must consider the weights, volumes, percent solids, and dilution factors when applying the 5x rule. These factors must be accounted for so that an actual comparison of the contamination is made. The Reviewer should be particularly aware of sample results which undiluted exceed the action level, but fall within the action level as a result of the subsequent dilution.

- If an associated method blank exhibits gross contamination, reject **[R 249]** positive results for the affected compounds.

  Note: The Functional Guidelines define gross contamination as saturated peaks. Professional judgment must be used to assess the impact the contamination has on the associated samples and which compounds are considered affected.

Action 2d: If an associated method blank was not analyzed for the samples, estimate **[J 249]** positive results.

Evaluation: ***The following item applies to validation only:***

Item 3: Recalculate one positive result per blank. Review the chromatograms and integration reports to evaluate blank results.

Action 3: If the calculated result does not agree within 5% or if a compound was misidentified, comment and assign reason code **[804]** to all applicable data. Review all other positive blank results.

2.12. Sample Preparation Raw Data

Review Items: Raw Data

Objective: To check that sample preparation raw data deliverable requirements have been met and that raw data are present in a form suitable for data assessment.

Sources: Attachment I to BOA Attachment 1, Base Methods

Evaluation: ***The following items apply to validation activities only:***

Item 1: Check that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:

- Analytical Batch identifier
- Date of preparation
- Identifiers for all samples, sample duplicates, and spikes
- Identifiers for at least one preparation blank and lab control sample
- For aqueous samples initial and final volumes for all samples and QC samples
- For solids and non-aqueous liquids reported by weight, initial weights and final volumes for all samples and QC samples

- For samples reported by weight, balance identifiers with dates of use.
- Dated signatures for at least one analyst and one reviewer

Action 1a: Check this item as complete if raw data were sufficient to perform calculations for all previous items.

Action 1b: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1c: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that instrument run logs are available for all analytical sequences.

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.13. TCLP Sample and Extract Preparation (Summary Form 2)

Review Items: Form 2 or equivalent, and raw data.

Objectives: To determine if samples were evaluated and prepared by the proper TCLP preparation method according to LIC, analyte, sample matrix, and analytical method utilized.

Sources: Attachment I to BOA Attachment 1, GR03 § 5, and Method 1311 for TCLP extraction.

Evaluation: ***The following Items apply to both verification and validation:***

Item 1: Check that a Form 2 or equivalent is present and the following information is included:

- Lab name, Lab Code, Analytical Batch Identifier and the RIN.
- Form 2 data for each sample.
- Physical descriptions of the samples (e.g. *multiphase liquid*, or *solids with no free liquid*) and a statement about which samples are of the same matrix.
- Result for the preliminary determination of percent solids and a description of the method of determination.
- An indication of whether particle size reduction was completed and how the reduction was completed, if reduction was required.
- A *Yes* or *No* to indicate whether free liquid was present in the sample.

- A *Yes, No, or N/A* to indicate whether any free liquid present was miscible with the extraction fluid.
- A volume recorded if a non-miscible liquid is present.
- A check that the preliminary evaluation of the pH of solids is recorded.
- A check that the evaluation of the pH of solids after the addition of acid is recorded, if applicable.
- A *Net Sample Weight (g)* or total weight of sample taken for the extraction process is recorded.
- A *Net Weight of Solids Extracted (g)* or the net weight of solids remaining after liquid solid separation is recorded.
- The type and weight of the extraction fluid added to the extraction vessel is recorded.
- The *Date and Time* of the start and end of the extraction period were recorded.
- The pH for the leachate solution after extraction and filtration, but before preservation was recorded.
- The method of preservation of the leachate was recorded.
- At least one spike-sample was prepared per waste type and analytical batch.
- At least one extraction blank was prepared per extraction fluid type and analytical batch.
- At least one duplicate sample was prepared per waste type and analytical batch.

Action 1a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 2: Determine that the appropriate TCLP Extraction method was completed for each sample.

Action 2: If the incorrect method was used for sample preparation and a CTR approved deviation was not documented, estimate **[J 207]** all applicable data.

Item 3: Check for evidence that samples with solids less than 0.5% were filtered as a TCLP Extract.

Action 3: If the percent solids is less than 0.5% and the sample was not filtered, estimate **[J 220]** positive results that exceed the regulatory level.

Item 4: Check for evidence of particle size reduction when the sample particle size exceeds 9.5 mm or the surface area is less than 3.1 cm^2.

Action 4: If particle size reduction is required and reduction was not performed, estimate **[J 222]** all sample results less than the regulatory level.

Item 5: Verify that TCLP results for extracts of samples with free liquids, both miscible and non-miscible, were reported appropriately.

Action 5a: If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was not addressed in the narrative, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 5b: If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was addressed in the narrative, comment and assign the reason code **[248].**

Item 6: Verify that the correct Extraction Fluid Type was used for the TCLP according to the following:.

- If the pH before or after (as applicable) the acidification is less than 5, Extraction Fluid Type 1 is to be used for the TCLP of all analyses.
- If the pH after acidification is greater than 5, Extraction Fluid Type 2 is to be used for the TCLP of all analyses.
- Extraction Fluid Type 1 is to have a pH of 4.93 ± 0.05
- Extraction Fluid Type 2 is to have a pH of 2.88 ± 0.05

Action 6a: If an incorrect or improperly prepared Extraction Fluid Type was used for the TCLP, comment and qualify using professional judgment, but qualify at a minimum as estimated **[J 233].**

Action 6b: If the extraction fluids are not numbered and cannot be identified from the data, comment and qualify using professional judgment, but qualify at a minimum as estimated **[J 224].**

Item 7: Verify that the correct amount of sample was processed for the TCLP.

Action 7: If the net sample weight processed for TCLP is less than 100 grams, use professional judgment to determine if the sample size is too small. Consider the physical state of the sample, the availability of sample, potential mixed waste issues (waste minimization priority), and whether particle size reduction was performed. At a minimum, comment and assign the reason code **[123].**

Item 8: Verify that the extraction period was within 16 to 20 hours.

Action 8: If the extraction start and end dates and times are not available or if the extraction time is not within 16-20 hours, use professional judgment to evaluate the data. Results near the regulatory limit may be biased low if the extraction time is less than 16 hours and results just above the regulatory limit may be biased high if the extraction time is greater than 20 hours. Results just below the regulatory limit that are suspected of low bias due to an insufficiently short extraction time are Rejected **[R 225].**

Item 9: Verify that TCLP Extracts were preserved appropriately, if analysis was not completed immediately.

Action 9: If the TCLP Extracts were not analyzed immediately after extraction and were not preserved at $4 \pm 2°$ C after extraction, comment and qualify all results less than the regulatory limit as estimated **[J 201].**

Item 10: Verify that a minimum of one TCLP Spike, Blank, and Duplicate are processed per waste type, preparation batch and extraction fluid type.

Action 10: If evidence of a spiked sample, duplicate sample, or extraction blank are not provided, comment and qualify all results as rejected **[R 168].**

Item 11: Verify that the ambient temperature during the extraction was maintained at 23 ± 2° C.

Action 11: If the ambient temperature during TCLP extraction was not maintained at 23 ± 2° C, estimate **[J 201]** all results less than the regulatory limit.

3. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report according to the General Data Assessment guidelines presented in DA-GR01. A Data Quality Assessment Report template for DV-SS03 is presented as Attachment 1.

4. REFERENCES

- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.
- Reason Codes for Data Assessment, Analytical Services Document
- Statement of Work for Analytical Measurements, General Laboratory Requirements, Module GR01-B.1, June 2, 1997.
- Statement of Work for Analytical Measurements, PCB/Pesticides, Module SS03-B, March 28, 1997.

ATTACHMENT 1: DATA QUALITY ASSESSMENT REPORT TEMPLATE

PEP

Data Quality Assessment Report

Rocky Flats Environmental Technology Site

RIN Number	Analytical Method/Analytical Specific Line Item Code		Review Level
Analytical Laboratory	**Assessment Performed by**	**Data Assessment Guideline Identifiers**	**Number of Samples**

Sample Numbers: _____

Quality Control Items	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, Narrative)		
Chain of Custody		
Holding Times		
Sample Preservation		
Surrogate Recovery		
MS/MSD Recovery		
Sample Results		
Calibration		
Analytical Sequence (CLP)		
Florisil Cartridge Check (CLP)		
Gel Permeation Chromatography (CLP)		
Blanks		
Sample Preparation		
EDD		
Other:		

Y Item was reviewed or non-compliance was identified
N Item was not reviewed or non-compliance was not identified
N/A Item is not applicable to the Line Item

PEP
Data Quality Assessment Report
Rocky Flats Environmental Technology Site

Data Assessment results are classified as either Action Items or Comments. Action Items are technical non-compliances that result in qualification of analytical results. Data may be qualified as valid (V), estimated (J), presumptively estimated (NJ), estimated at an elevated level of detection (UJ), or rejected (R). Multiple qualifiers may be associated with any given data point based on the number of problems identified, however, the assigned qualifier is based upon the following hierarchy: R, UJ, NJ, J, V. All data points that are not qualified based upon action items in this report are considered valid (V). Comments are technical non-compliances or contractual non-compliances that do not result in qualification of data.

Action Items:

Comments:

Verification/Validation Signature_____ Date:_____

Reviewer Signature_____ Date:_____
(*Validation Only*)

33/33